Encapsulation of Atrazine Within a Starch Matrix by Extrusion Processing

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ABSTRACT

Atrazine, a powdered herbicide widely used in agriculture, was encapsulated within a starch matrix by a continuous process in a twin-screw extruder under various conditions. Four extrusion procedures were evaluated. Unmodified cornstarch was gelatinized in the extruder in the presence of the herbicide and water (starch concentrations in the water were 20–65%); atrazine was added at levels of 5–20% (product basis). The extruded materials were cooled, dried, ground, and sieved to obtain granular products of 10–20 and 20–40 mesh. These products were analyzed for encapsulation efficiency, swellability in water, and release of active agent in aqueous ethanol (10%). Effects of extrusion, milling, and drying variables on properties of encapsulated products were studied. This work suggests that excellent potential exists for producing starch-encapsulated products with a range of customized release properties by applying twin-screw extruder technology. The production rate of the starch-encapsulated herbicide containing 10% atrazine was scaled up from 12 lb/hr (db) in a ZSK 30 to 167 lb/hr in a ZSK 57 twin-screw extruder. An encapsulation-efficiency value of about 90% was achieved at both production rates. The starch-encapsulated atrazine, prepared in the ZSK 57, will be evaluated in field trials in seven states.

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Pesticides have been encapsulated within starch matrices by various techniques to control rate of release, decomposition, leaching, groundwater contamination, dermal toxicity, and other problems associated with the use of these active agents (Shasha 1980, Schreiber and Shasha 1988). Successful starch encapsulation techniques investigated at our research center include ionic and covalent cross-linking of starch xanthate, coagulation of alkali-treated starch with chemicals such as calcium chloride or boric acid, and retrogradation of starch without chemicals (Shasha et al 1981, 1984; Wing and Otey 1983; Wing et al 1987). The starch-retrogradation technique is attractive for encapsulating various materials for agricultural, feed, food, medicinal, and other uses because the procedure is simple and economical, and it eliminates the use of cross-linking chemicals.

The starch-retrogradation procedure for encapsulating pesticides (or other materials) involves gelatinization of the starch, blending the pesticide immediately into the hot gelatinized starch, and allowing natural retrogradation to occur as the mixture cools and dries. Retrogradation is the physical process by which hydrated or soluble starch molecules reassociate and revert to water-insoluble forms. The rate and extent of retrogradation are affected by many factors, such as the starch amylopectin-amylose ratio (about 3:1 for ordinary cornstarch); molecular weight of the starch fractions; concentration of other materials in the system; and dispersion, cooling, and drying conditions.

Bioactive agents have been incorporated into starch matrices by batch methods with limited processing flexibility, control, and efficiency. We recently found that the encapsulation can be accomplished rapidly, efficiently, and continuously over a wide range of conditions using a corotating, fully intermeshing, twin-screw extruder. Twin-screw extruders are well recognized in the food, feed, plastics, and other industries as highly versatile mixers-reactors for the most difficult continuous processing requirements (Sneller 1985, Frund 1986, Mielcarek 1987), and we believe a wide range of customized starch-encapsulated products can be developed most readily with such extruders.

The present work describes our initial study, in which atrazine—a common agricultural herbicide—was encapsulated within cornstarch matrices by twin-screw extrusion. The effects of processing variables on encapsulation efficiency, swellability, and rate of herbicide release were investigated.

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MATERIALS AND METHODS

Extrusion Processing Materials

Materials included unmodified cornstarch (Buffalo 3401, CPC International, Englewood Cliffs, NJ), powdered atrazine (2-chloro-4-ethylamino-6-isopropylamino-*s*-triazine, technical grade, 97% active agent, CIBA-GEIGY Corp., Greensboro, NC), and house-supplied distilled water.

Extrusion Equipment

The extruder was a ZSK 30 twin-screw extruder with a 15-hp DC drive and screw speeds of 50–500 rpm (Werner and Pfleiderer Corp., Ramsey, NJ). The processing section included 14 individual barrel sections (BS) (each 90 mm long), one barrel spacer (30 mm long), and an end plate (30 mm long), coupled by four tie rods. Barrel bore and screw diameters were 30.85 and 30.70 mm, respectively. The ratio of the barrel length to the screw diameter was 43:1.

Each BS except the first had a 500-watt electric heater. Barrel-temperature thermocouples were located at BS 3, 5, 7, 9, 11, 12, and 14. Material-temperature thermocouples were located at BS 9 and at the die-head assembly, which was equipped with a 1,500-watt heater. Chilled water, supplied by a chiller (model WC 7.5 ME/XT, Application Engineering Corp., Wood Dale, IL), was circulated through the extruder barrel jackets, which were equipped with solenoid valves and return-line check valves to regulate barrel temperature.

The twin screw was composed of 67 individual slip-on screws for feeding, conveying, mixing, and pumping (Carr and Cunningham, 1989). The die-head assembly (zone 8), with a discharge opening of 8 × 40 mm, was not equipped with a die. Starch and atrazine were fed into the extruder by single-screw and twin-screw volumetric feeders (models S-200 and T-20, respectively, K-TRON Corp., Pitman, NJ). Water was injected through drilled thermocouple ports by reciprocating, positive-displacement piston pumps (models EK-3 and FC-3/V, American LEWA, Inc., Holliston, MA). Feed rates, screw speed, and barrel temperatures were controlled from a remote panel (Werner and Pfleiderer Corp.)

Extrusion Procedures

Four procedures were used (Table I). The screw speed was 400 rpm, and the starch feed rate was 84 g/min (75.6 g/min, db). The percent of atrazine addition was calculated as (g of atrazine) ÷ (84 g + g of atrazine) × 100.

Procedure 1. The cornstarch (10% moisture) and herbicide were preblended to give 5, 10, 15, and 20% atrazine. Each blend was fed into BS 1, while water was pumped into BS 3 at 24–132 ml/min to give starch concentrations of 35–65%. Also, water was fed at 10 ml/min to give a concentration of 70% starch with 5% atrazine. Starch concentrations were calculated as

(g of starch, db) ÷ (g of starch with 10% moisture + g of water added) × 100. Barrel temperatures were 25°C (BS 1), 70°C (BS 2 and 3, zone 1), 90°C (BS 3 and 4, zone 2), 100°C (BS 5–10, zones 3–5), 90°C (BS 11–14, zones 6 and 7), and 90°C (die head, zone 8). All sections except BS 1 were closed to the atmosphere.

Procedure 2. A preblend of starch and atrazine (5%) was fed into BS 1, and water was fed into BS 3, to give an initial starch concentration of 35%. Additional water was fed into BS 7 to obtain a final starch concentration of 20%. Barrel temperatures were 80, 100, 125, 125, 100, 100, 80, and 80°C for zones 1–8, respectively.

Procedure 3. A preblend of starch and atrazine was fed into BS 1, and water was fed into BS 3, to give an initial starch concentration of 55%. Additional granular starch was fed into BS 11 to give a final starch concentration of 65% and a final atrazine addition level of 5% (procedure 3a). The same method was used to obtain an initial starch concentration of 35% at BS 3 and a final starch concentration of 65% at BS 11 (procedure 3b, 2.5% atrazine). Barrel temperatures in both cases were 70, 90, 100, 100, 50 (BS 10 and 11), 70, 90, and 90°C for zones 1–8, respectively.

Procedure 4. Starch (10% moisture) alone was fed into BS 1, and water was fed into BS 3, to give final starch concentrations of 35 and 65%. Atrazine alone was fed into BS 11. In procedure 4a, barrel temperatures were 70, 90, 100, 90, 50 (BS 10 and 11, atrazine addition), 70, 90, and 90°C for zones 1–8, respectively. In procedure 4b, temperatures were 70, 90, 100, 90, 50 (BS 10 and 11, atrazine), 30, 30, and 30°C for zones 1–8, respectively.

Scale-up. The encapsulation process was scaled up on a 57-mm twin-screw extruder (ZSK 57) of the same type and design as the 30-mm extruder (ZSK 30), except that the ratio of the barrel length to the screw diameter was 30 rather than 43.

In the scale-up, starch was fed into BS 1 at 150 lb/hr (135 lb, db), atrazine (10% level of addition) was fed into BS 1 at 16.7 lb/hr, and deionized water (70% starch concentration) was fed into BS 2 at 42.9 lb/hr. The die-head assembly was equipped with a die that had 20 holes 5 mm in diameter. Barrel temperature was 25°C at BS 1, 75°C at BS 2–4, and 95°C at BS 5–10. The screw was a less severe mixing screw than the one used in the original experiment and was composed of alternating conveying and kneading block elements in the starch gelatinization zones. Screw speed was 200 rpm. The extrudate was air-dried, milled, sieved, and analyzed essentially as described for the materials extruded in the ZSK 30 extruder.

Sample Handling

Extrudate samples weighing about 250–550 g (db) were stored overnight in a refrigerator at 34–40°F, dried under a hood at ambient conditions, and ground in a disk mill. The milled samples were allowed to equilibrate to about 7–10% moisture and then were sieved to obtain granular products of 10–20 and 20–40 mesh.

Analysis

Encapsulation efficiency. Sieved samples were washed three times with chloroform to remove atrazine that was exposed in grinding. Kjeldahl nitrogen was run on the unwashed samples to quantify total atrazine and on the washed samples to quantify encapsulated atrazine.

Swellability. A 200-mg portion of each sample was placed in a 10-ml graduated cylinder, to which 4.0 ml of distilled water (30°C) was added. The mixture was gently stirred several times during the first 3 hr to prevent clumping. After 24 hr, the percent of increase in volume of the swollen sample was calculated.

Atrazine release. Portions of the chloroform-washed samples (84–422 mg) containing 10.0 mg of atrazine and 75 ml of aqueous ethanol (10% ethanol, v/v) were placed in 125-ml Erlenmeyer flasks and agitated on an orbital shaker (Lab-Line, Melrose Park, IL) at 250 rpm for up to 72 hr. Solvent volume was 20% in excess of that required to dissolve the total atrazine in each sample. The use of aqueous ethanol provided a convenient and rapid laboratory procedure for comparing release of atrazine from the various products. After each treatment period, the available atrazine was extracted with chloroform (25 ml). Atrazine concentration was measured with a gas-liquid chromatograph (Tracon 560, Austin, TX) with a Chromosorb WHP column. A standard curve was made with trifluralin as an internal standard.

RESULTS AND DISCUSSION

Extrusion Procedures

In procedure 1, preblends of granular atrazine and starch (10% moisture) were fed into BS 1, water was fed into BS 3, and the mixtures were processed at starch concentrations of 35–65% with atrazine addition levels of 5–20%. This procedure, which required the least equipment and the fewest control measures, produced the most uniform product.

In procedure 2, preblended starch and atrazine were fed into BS 2, and processed water was split between BS 3 and 7 to obtain a final starch concentration of 20%. Water was fed into BS 7 to prevent back-flooding into BS 1. The barrel temperature in zones 3 and 4 (BS 6–9) was increased to 125°C to fully gelatinize the starch in this relatively dilute system.

Procedure 2 was the least energy efficient because more water had to be heated in the extruder and removed before milling the extrudate.

In procedure 3, preblended starch and atrazine were fed into BS 1, water was fed into BS 3 to give starch concentrations of 55% (procedure 3a) and 35% (procedure 3b), and additional granular starch alone was fed into BS 11 to obtain a final starch concentration of 65%. Atrazine addition levels were 5 and 2.5%, respectively. This procedure required an additional dry-solids volumetric feeder but provided some benefits. The extrudates were more easily crumbled and milled because the starch added to BS 11 remained primarily in granular form within the product

TABLE I
Extrusion Procedures for Encapsulation[a]

Procedure No.	Material Added Position (Barrel Section No.)			Extrudate			Barrel Temperature (°C) Zone No.							
	Starch	Atrazine	Water	Starch Concentration (%)[b]	Atrazine (%)	Total Solids (%)	1	2	3	4	5	6	7	8
1[c]	1	1	3	35–70	5–20	36–75	70	90	100	100	100	90	90	90
2	1	1	3,7	20	5	21	80	100	125	125	100	100	80	80
3a[d]	1, 11	1	3	65	5[e]	66	70	90	100	100	50	70	90	90
3b[d]	1, 11	1	3	65	2.5[f]	66	70	90	100	100	50	70	90	90
4a	1	11	3	35, 65	5	36, 66	70	90	100	90	50	70	90	90
4b	1	11	3	35, 65	5	36, 66	70	90	100	90	50	30	30	30

[a] Using a ZSK 30 twin-screw extruder (see materials and methods section in text).
[b] Starch concentration = g of dry starch ÷ (g of starch with 10% moisture + g of added water) × 100.
[c] Starch + atrazine preblend. Starch concentration was 35–65% for 5–20% atrazine and 70% for 5% atrazine.
[d] Starch + atrazine addition at BS 1 plus starch alone at BS 11.
[e] Starch concentration = 55% at BS 3-10 and 65% from BS 11 through last zone.
[f] Starch concentration = 35% at BS 3-10 and 65% from BS 11 through last zone.

matrix. Also, this procedure provided a unique means for altering the physical properties of the starch matrix (discussed later).

In procedure 4, atrazine was fed downstream into the gelatinized starch at BS 1 (35 and 65% starch concentrations). This procedure is probably preferable for additives that are sensitive to heat and shear. However, atrazine per se was quite stable under all the extrusion conditions studied. This procedure would also eliminate any inhibiting effect an additive might have on starch gelatinization.

The milled products were analyzed for encapsulation efficiency (EE), swellability, and release rate. In this work, EE is defined as (g of atrazine in the chloroform-washed product) ÷ (g of atrazine in the unwashed product) × 100. Chloroform extracted atrazine from the surface of the product granules without significantly extracting atrazine from the interior of the granules.

EE was not appreciably affected by differences in the four extrusion procedures. Swellability of the products soaked in water were similar when prepared by procedures 1, 2, and 4, whereas products prepared by procedure 3 swelled to relatively greater extents. The release rate of atrazine from the products in aqueous ethanol (10%, v/v) was affected primarily by differences between procedures 1 and 3 and by variables within each of these procedures. Thus swellability, release rate, and EE are discussed in the following sections for procedures 1 and 3, but primarily for procedure 1.

EE

Figure 1 shows that EE was not appreciably affected by processing the mixtures of aqueous starch and atrazine in the extruder by procedure 1 at starch concentrations of 35–65% for 5–20% atrazine addition levels and at 70% starch concentration for 5% atrazine addition (milled products of 10–20 mesh). The level of atrazine addition had a rather small effect on EE.

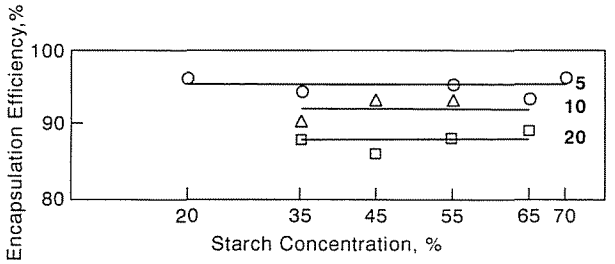

Fig. 1. Efficiency of encapsulating atrazine in cornstarch matrices by extrusion processing with procedure 1 (20–40 mesh products). Atrazine addition levels were 5, 10, and 20%, product weight basis. Data indicated by the circle at 65% starch concentration belongs with the data on the top line; no data were obtained at 65% starch concentration for the 10% level of atrazine addition.

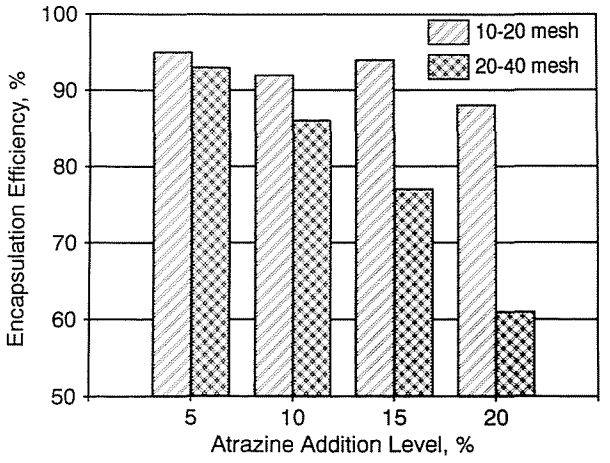

Fig. 2. Effect of particle size and atrazine addition level on starch encapsulation efficiency using procedure 1.

Disregarding starch concentration, average EE values were 95, 92, and 88% for atrazine addition levels of 5, 10, and 20%, respectively.

The level of atrazine addition and the particle size of the milled products appreciably affected EE (Fig. 2). For example, when atrazine was increased from 5 to 20%, EE decreased from 93 to 61% for the 20–40 mesh products, compared with a decrease from 95 to 88% for the larger 10–20 mesh products. Particle size and particle-size distribution could be controlled and targeted to meet specific atrazine release needs in field application. Moisture content of the extrudate at the time of milling also affected EE values. Limited data indicate that the extrudates should be dried to about 8–10% moisture before milling for best results (Table II). Further study is needed to see if milling the extrudates at various levels of moisture content could be used to control initial herbicide dose rates in field applications. An opposite effect regarding moisture content before milling was reported by Stout et al (1979) for cross-linked starch-encapsulated herbicides prepared by a batch process. Excellent EE was obtained at extruder-material temperatures as low as 78°C (data not shown), but further study is needed.

Swellability

The extent to which the products with starch-encapsulated atrazine swelled when soaked in water for 24 hr at 25–27°C are shown in Figure 3 (procedure 1, 20–40 mesh products). These data show that the products swelled more as starch concentration

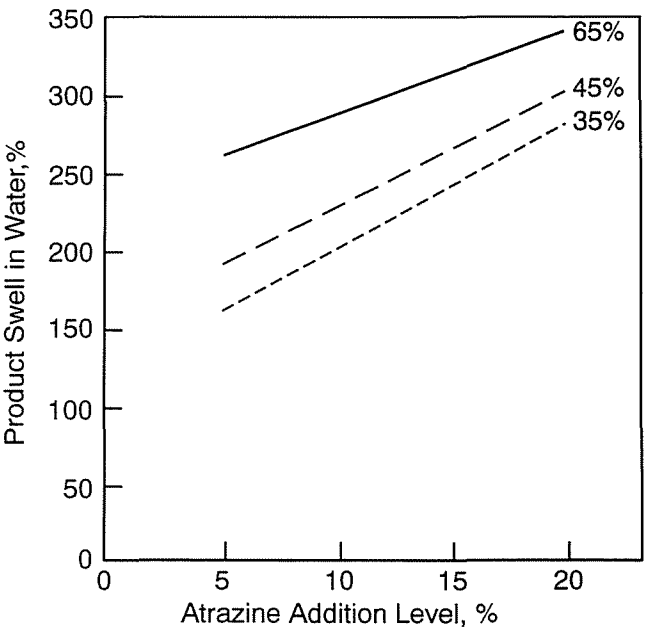

Fig. 3. Effect of starch concentration and atrazine addition level on swellability of starch-encapsulated products in water. Starch extrusion was processed at 35–65% starch concentrations; milled products (20–40 mesh) were soaked for 24 hr in water at 25–27°C (using procedure 1).

TABLE II
Effect of Wet and Dry Milling of Extrudate on Encapsulation Efficiency

Moisture Content of Extrudate[a] (%)	Milled Particle Size (Mesh)	Encapsulation Efficiency[b] (%)
8	10–20	96
25	10–20	90
8	20–40	87
25	20–40	73

[a] Extrudate was produced at 65% starch concentration by procedure 1 and allowed to dry to levels of moisture contents shown before milling.
[b] Analysis of encapsulation efficiency was run after the milled particles were air-dried to about 8–10% moisture.

TABLE III
Release of Atrazine from Starch-Encapsulated Product in Aqueous Media[a]

Level of Addition (%)	Atrazine Encapsulated		Atrazine Released					
			Percent			(g/100 g of product)		
	(%)	(g/100 g of product)	(4 hr)	(24 hr)	(72 hr)	(4 hr)	(24 hr)	(2 hr)
5	94	4.7	21	29	32	1.0	1.4	1.5
10	87	8.7	20	30	41	1.7	1.6	3.6
20	61	12.2	18	29	35	2.2	3.5	4.3

[a]Chloroform-washed products (20–40 mesh) in aqueous 10% ethanol. Extrusion procedure 1 (65% starch concentration).

TABLE IV
Release of Atrazine from Starch-Encapsulated Product in Aqueous Media[a]

Starch Concentration (%)	Atrazine Released in 24 hr		Product Swell in Water (%)
	(%)	(g/100 g of product)	
35	23	1.1	160
45	25	1.2	190
55	27	1.3	200
65	33	1.5	260

[a]Level of atrazine addition = 5%, procedure 1.

was increased from 35 to 65% and as the atrazine addition level was increased from 5 to 20%. Products swelled 160 to 340% for the conditions shown. In previous work on starch encapsulation of herbicides by retrogradation of jet-cooked starch, an opposite swelling trend occurred with respect to starch concentrations of 5–33% (Wing et al 1987). In that work, gelatinized starch and atrazine were blended in a Sigma blade mixer.

In data not shown, the greatest swelling (580%) occurred when dry granular starch was added to the gelatinized starch at BS 11 by procedure 3b. The granular starch probably functions as a filler to reduce the extent of retrogradation that occurs when the gelatinized starch is cooled and dried. Presumably, similar results might be obtained by incorporating clay, silica, carbonates, or other fillers into the gelatinized starch. Products with controlled release rates might be produced in this way. In a study that is underway, the final products swelled more in water when high-solids mixtures in the extruder were extruded through a die. For example, a 20–40 mesh product swelled 440% when the mixture (65% starch concentration, 5% atrazine) was extruded through a die with two holes 4 mm in diameter, versus 260% without a die. Thus, product properties might be tailored by extruding mixtures through dies at various conditions of temperature, pressure, and shear.

Release Rate

Table III shows data on release rate for mesh products of 20–40 prepared by procedure 1 at 65% starch concentration with 5–20% atrazine additions. The chloroform-washed products were mildly swirled in aqueous ethanol for up to 72 hr (described in materials and methods section). These data show that percentages of atrazine released from the product within each time period were similar for all levels of atrazine addition. For example, about 20% of the atrazine was released in 4 hr for products with either 4.7 or 12.2 g of atrazine per 100 g of product. The absolute amounts released from the products, of course, varied considerably. Small but significant increases in release rates occurred as starch concentration was increased from 35 to 65% (Table IV). The quantities released in 24 hr increased from about 1.1 to 1.5 g/100 g of product. These data indicate that release rate and swellability of the products in water are directly related, and both increase with greater starch concentration.

The data in Figure 4, which compares the release rate of 20–40 mesh products prepared by procedures 1 and 3, show that when the starch concentration of gelatinized starch in the extruder was increased from 35 to 65% by addition of the granular starch to BS 11, the percentages of atrazine released were about 1.5–2.5 times greater for procedure 3a than for procedure 1 (20–40-mesh product). Swellability increased correspondingly. Although the

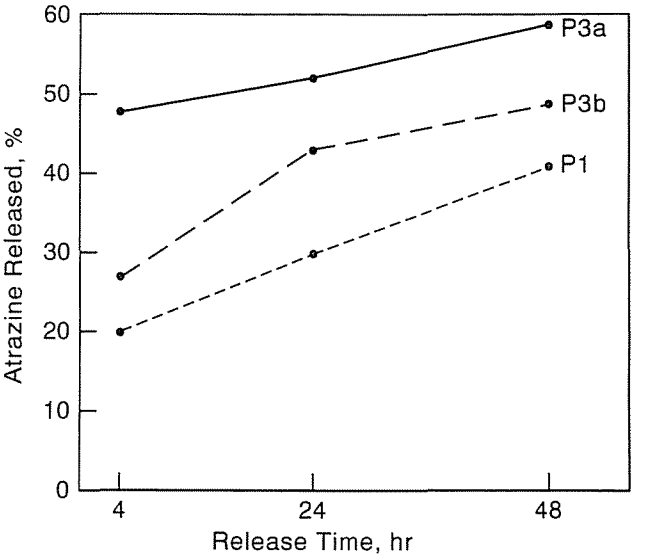

Fig. 4. Release rate of starch-encapsulated atrazine products in aqueous 10% ethanol. Products of 20–40 mesh were mildly agitated at 25–27°C. Procedures 1, 3a, and 3b were used, with 65% starch concentration and 5% atrazine for procedures 1 and 3b and 2.5% atrazine for procedure 3a.

atrazine addition level was 2.5% for procedure 3a versus 5% for procedures 1 and 3a, the general trend was that atrazine release increased as the level of addition increased. Therefore, the effect of adding granular starch as a filler downstream in procedure 3a would be at least as pronounced with 5% atrazine as with 2.5%.

Studies of the release rate were performed in aqueous ethanol only to provide a convenient and rapid laboratory comparison of the various products. Also, in field application, release of atrazine by water is much slower than by aqueous ethanol, which is used in the laboratory for convenience (see materials and methods section). This is true even for products containing as little as 5% atrazine. Also, although chloroform extracts several percentage points of the atrazine from the dry granules, very little can be extracted with chloroform when the granules first swell in water. It appears that upon hydration, atrazine on or near the surface becomes trapped by the swollen matrix. Also, water may act as a barrier to encountering chloroform. Studies of the products are underway in greenhouses and field plots at several locations.

Scale-up of Extrusion Encapsulation and Studies in Progress

The encapsulation process was scaled up as described in the materials and methods section. Table V shows that atrazine was encapsulated as effectively in the scale-up at 167 lb of product per hr as at 12 lb/hr in the ZSK 30 extruder. However, the product from the larger scale production swelled more and released atrazine at a faster rate in aqueous ethanol. This was probably because the material was extruded through a die, whereas no die was used for preparing the products in the ZSK 30 extruder, except in more recent work (previously noted).

Our extrusion studies have been expanded to include the

TABLE V
Properties of Starch-Encapsulated Product from Scaled-up Production

Extruder (mm)	Production of Product (lb/hr)[a]	Atrazine Encapsulated (%)	Swelling in Water (%)[b]	Atrazine Released, %		
				(In 4 hr)	(In 24 hr)	(In 72 hr)
57	167	89	380	26	40	61
30	12	90	220	20	30	41

[a]Production rate is based on final product with 10% moisture.
[b]Product (20–40 mesh) was soaked for 24 hr in water at 25–27°C.

encapsulation of various types of liquid, crystalline, and granular herbicides within waxy, high-amylose, and ordinary cornstarch substrates under a broader range of processing conditions. New, more comprehensive methods for analysis and evaluation of the products are being developed. Scale-up studies for starch encapsulation of liquid and crystalline herbicides are underway. The products will be evaluated in field trials.

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